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CENTRAL INTELLIGENCE AGENCY
INFORMATION FROM
FOREIGN DOCUMENTS OR RADIO BROADCASTS

REPORT
CD NO. [redacted]

STAT

COUNTRY USSR

SUBJECT Scientific research

DATE OF INFORMATION 1947

HOW PUBLISHED Periodicals

DATE DIST. 21 January 1949

WHERE PUBLISHED USSR

NO. OF PAGES 2

DATE PUBLISHED 1947

LANGUAGE Russian

SUPPLEMENT TO REPORT NO.

THIS IS UNEVALUATED INFORMATION

SOURCE Documentary as indicated. (Information requested.)

RECENTLY PUBLISHED RESEARCH OF THE LENINGRAD ENGINEERING ECONOMY INSTITUTE IMENI V. M. MOLOTOV

"Thermal Decomposition of Ester Chlorides of Silicic Acid," Yu. N. Vol'nov, Leningrad Eng Econ Inst imeni V. M. Molotov

"Zhur Obshch Khimii" Vol 17, 1947, pp 1428-35

Generally, stability increases with increased molecular weight and decreases from n-esters to iso-esters. Aromatic esters are more stable than aliphatic. $EtOSiCl_3$ (50 g), heated to 100° 2 hours and distilled, gave, beside the unchanged material (75%), 7 g $SiCl_4$ and 2.7 g $(EtO)_2SiCl_2$. When $EtOSiCl_3$ was refluxed 4 hours, gas evolution was noted (identified as EtCl), while distillation of the residue gave 50% unchanged material, about 5 g $SiCl_4$, and 3 g $(EtO)_2SiCl_2$. $(EtO)_2SiCl_2$ was uncharged after 2 hours at 100° or refluxing 4 hours. $(EtO)_3SiCl$ (80 g), heated to 100° 2.5 hours, gave 3 g $(EtO)_2SiCl_2$, 63 g starting material and about 1.5 g $(EtO)_4Si$. When $(EtO)_3SiCl$ (30 g) was refluxed 6-7 hours, there was obtained essentially 100° disproportionation: 2.5 g Et_2O, 1 g $SiCl_4$, a small amount of $(EtO)_2SiCl_2$, 12 g $(EtO)_4Si$, and 40 g residue, from which it was possible to isolate some $(EtO)_6Si_2O$, b$_{75}$ 150-70°. $C_6H_{13}OSiCl_3$ (20 g) after 4 hours at 100° gave 1.1 g $SiCl_4$ and 4 g $(C_6H_{13}O)_2SiCl_2$, besides 87-94% starting material. $C_8H_{17}OSiCl_3$ was unchanged after heating to 100°, but on refluxing 6 hours there were obtained from 13 g starting material 2.5 g crude $SiCl_4$, 2 g $(C_8H_{17}O)_2SiCl_2$, and 2 g tar; the rest was unchanged starting material. $(C_8H_{17}O)_3SiCl$ is unchanged on

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heating to 100°, but after refluxing as above gave $C_8H_{16}O$, b 124-6°, tar, and $(C_8H_{17}O)_2SiCl_2$, beside the starting material; no tetraalkyl derivative was isolated. $PhCSiCl_3$ (30 g) refluxed 5 hours gave a trace of $SiCl_4$, 28 g unchanged material, and about 1 g crude $(PhO)_2SiCl_2$. $(PhO)_3SiCl$, refluxed 5 hours, with continuous collection of low-boiling products gave 0.2 g $SiCl_4$, 0.3 g $(PhO)_2SiCl_2$, 35 g unchanged material, and 2 g $(Pho)_4Si$. Thymyltrichlorosilane (20 g) after refluxing 5 - 6 hours gave $SiCl_4$, unchanged material, and dithymyldichlorosilane. o-Methoxyphenyltrichlorosilane (30 g) refluxed 4 hours gave 0.8 g $SiCl_4$, unchanged material, bis- (o-methoxyphenyl) dichlorosilane (undistillable resin), and MeCl, besides a crystalline solid, isolated on standing, from material b 240-320°; the solid (no mp or yield given) is apparently a cyclic phenylenedioxydichlorosilane, as a result of loss of MeCl in an intramolecular reaction.

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